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We have continued the development and use of the electrical potential method for measuring the length of the crack and confirmed that this method gives accurate and reliable data. We have completed the fatigue testing of the aluminium-alloy substrates (BS 5083 grade), pretreated using various surface treatments, and bonded with the 'AF 163' film adhesive. The 'hot/wet' fatigue tests clearly reveal the effect of an aggresive, hostile environment on the mechanical performance of adhesive joints and may provide the basis for a very effective accelerated ageing test method.

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1. INTRODUCTION

The bonded joints employed were tapered-double cantilever beam (TDCB) specimens, which consist of aluminium alloy substrates (BS 5083 grade), pretreated using various surface treatments, and bonded with '3M AF 163' film adhesive. The details of the preparation of the TDCB joints were reported previously [1].

2. OVERALL PROGRESS

The work has progressed well and in the last six months we have:

(i) Continued the development and use of the automatic method for monitoring the rate of crack growth. This is based upon an electrical potential method for measuring the length of the crack.

(ii) Confirmed that the above method gives accurate and reliable data when employed to measure the length of the propagating crack in water at 28°C, as well as in a relatively dry environment. This has been proven by comparing the measurements from the electrical potential method with those from visual observations using a travelling microscope.

(iii) Started to employ another method for measuring the length of the propagating crack. This method is based upon using an ultrasonic probe which locates the tip of the growing crack. This method is particularly useful for samples where the crack cannot be easily seen from the side of the specimen and where long exposure times in water, or other environments, are required. Initial testing is about to begin using this method.

(iv) We have completed the fatigue testing of the aluminium-alloy substrates (BS 5083 grade), pretreated using various surface treatments, and bonded with '3M AF 163' film adhesive.

(v) The test conditions for the fatigue tests have been:

(a) Laboratory atmosphere of 55% r.h. and 25°C.

(b) Immersion in water at 28°C.

(vi) The pretreatments used for the aluminium alloy have been:

(a) Chromic acid etched (CAE).

(b) Phosphoric acid anodised (PAA).

(c) Phosphoric acid anodised and primer '3M EC-3924B' (Primer).

(vii) Specimens for the testing of bonded fibre-composite materials have been prepared.

3. EXPERIMENTAL DETAILS

The compliance, dC/da, of the TDCB specimens as a function of crack length is given by:

$$\frac{dC}{da} = \frac{8m}{Eb} \tag{1}$$

where E is the modulus of the substrate, b is the width of the specimen, C is the compliance (C= displacement (δ)/load (P)) and m is the specimen contour factor given by:

$$m = \frac{3a^2}{d^3} + \frac{1}{d} \tag{2}$$

where d is the height of the contoured beam at a crack length a.

The fatigue behaviour at a frequency of 5 Hz and a displacement ratio, δ_{ratio}, ($\delta_{ratio} = \delta_{min}/\delta_{max}$) of 0.5 has been ascertained. The data have been plotted in the form of the rate of crack growth, da/dN, per cycle versus the maximum applied strain energy release rate, G_{max}, in each cycle. Where the value of G_{max} is given by:

$$G_{max} = \frac{P_{max}}{2b} \cdot \frac{dC}{da} \tag{3}$$

where P_{max} is the maximum load applied to the specimen per cycle. As described in the previous report [2], the 'Mac Lab' unit is connected to a 'Macintosh PC'. The PC acquires the change in crack length as a function of the time (i.e. number of cycles) and a computer program, based on the ASTM Method E647-88, calculates the rate of crack growth per cycle, da/dN. The PC also acquires the signals of the maximum load and displacement being applied to the specimen, and therefore the corresponding value of G_{max} may be deduced from equation (3).

4. RESULTS

4.1 Static fracture behaviour

As discussed previously [2], the aluminium alloy beams were subjected to a chromic-acid etch pretreatment and then bonded using the 'AF 163' adhesive. The static value of the adhesive fracture energy, G_c, (also termed the 'critical strain energy release rate') for a test rate of 1 mm/min was determined was found to be 1720 J/m^2. The locus of failure was cohesive through the adhesive layer.

4.2 Fatigue fracture behaviour

4.2.1 Chromic-acid etched (CAE) pretreatment

The aluminium-alloy beams were subjected to a chromic-acid etch (CAE) pretreatment and then bonded using the 'AF 163' adhesive. The fatigue crack growth behaviour was determined at a frequency of 5 Hz and a displacement ratio of 0.5. For the 'dry' tests, the test temperature was 25°C and the relative humidity was 55% r.h. The results are shown in Figure 1, and are represented by the points shown as '+'. In all cases the locus of failure was cohesive through the adhesive layer. A threshold value of the adhesive fracture energy, G_{th}, from the cyclic fatigue tests is indicated in Figure 1. The value of the threshold value of the adhesive fracture energy, G_{th}, represents the value below which fatigue failure in the given environment would not be observed. From Figure 1, the value of G_{th} in a 'dry' environment is about 550 J/m^2. This represents approximately 30% of the initial static value which was obtained; i.e. 1720 J/m^2.

The above experiments were then repeated in the 'wet' environment of immersion in water at 28°C. The results are also shown in Figure 1. Several interesting points emerge. Firstly, for the experiments conducted at a relatively high maximum displacement (δ_{max} values of 2.6 mm) the locus of failure was again via cohesive fracture through the adhesive layer. However, the fatigue data (shown by the 'o' points) are significantly inferior to those from the above 'dry' tests. These observations are suggested to arise from this high maximum displacement causing the fatigue test to be finished in a very short time, before the water could attack the interface. In this case the slight difference in the 'wet' compared to the 'dry' results is due to plasticisation of the adhesive at the crack tip by the ingressing water. Secondly, however, when lower values of the maximum displacement (δ_{max} values of 1.4 and 1.6 mm) were used, then the experiments lasted significantly longer in time. In these cases <u>interfacial</u> failure was observed, and the fatigue behaviour of the 'wet' tests was greatly inferior to that shown by the 'dry' tests. Thus, in these joints, the water attacked and weakened the interfacial regions. This is reflected in the value of G_{th} being about 50 J/m^2. (Recall that the initial static value which was obtained was 1720 J/m^2. Hence, this 'wet' value of G_{th} is only about 3% of the initial static value!)

4.2.2 Phosphoric acid anodising (PAA) pretreatment

The above experiments were repeated using either (i) a PAA pretreatment or (ii) a PAA pretreatment followed by applying the 'EC-3924B' primer. It was found that upon conducting the experiments in water at 28°C that at all values of the maximum displacement, δ_{max}, the locus of failure remained cohesive in the adhesive layer. Further, there was no major decrease in the fatigue behaviour, as was the case for the majority of the CAE treated joints, see above. However, there was a small decrease in the fatigue resistance of the 'wet' tests conducted on the PAA treated joints, again presumably due to water plasticisation at the crack tip. The value of G_{th} is about 320 J/m^2. This represents approximately 20% of the

initial static value which was obtained; i.e. 1720 J/m^2. (For comparison, the 'dry' value of G_{th} was about 550 J/m^2, see above.) The above observations apply equally to whether the treatment used was either (i) a PAA pretreatment or (ii) a PAA pretreatment followed by the 'EC-3924B' primer.

Thus, the 'hot/wet' fatigue tests clearly can reveal the effect an aggressive, hostile environment may have upon the mechanical performance of an adhesive joint, and may provide the basis for a very effective accelerated ageing test. Further, it appears that a threshold value of the strain-energy release rate, G_{th}, does exist and may be used to rank the fatigue limit behaviour of different adhesive systems and their resistance to hostile environments. Finally, the use of either (i) a PAA pretreatment or (ii) a PAA pretreatment followed by the 'EC-3924B' primer for the aluminium-alloy substrates bonded with the 'AF163' adhesive leads to excellent joint durability.

5. FUTURE WORK

The future work planned is:

(i) To examine the fatigue crack growth behaviour of carbon-fibre thermoplastic composites bonded using the 'AF163' adhesive. The composites will be carbon-fibre/poly(ether-ether ketone) and glass-fibre/poly(phenylene sulphide) materials, and they will be corona treated prior to bonding.

(ii) To examine the fatigue crack growth behaviour of carbon-fibre thermosetting composites bonded using the 'AF163' adhesive. The composite will be a carbon-fibre/epoxy and will be subjected to a light abrasion and solvent wipe prior to bonding.

(iii) To also use a typical hot-cured past adhesive for the above studies.

(iv) To continue to explore various methods for determining the crack length, and hence accurately ascertain the value of da/dN and the value of the threshold value, G_{th}.

(v) To explore further the use the fatigue test method, and the use of the values of G_{th}, as a sound basis for ranking the performance of adhesive joints under hostile environments.

6. REFERENCES

[1] A.J. Kinloch and M. Fernando, '2nd Interim Report', US Army, ERO, June 1993.

[2] A.J. Kinloch, M. Fernando and P. Lam, '3rd Interim Report', US Army, ERO, December 1993.

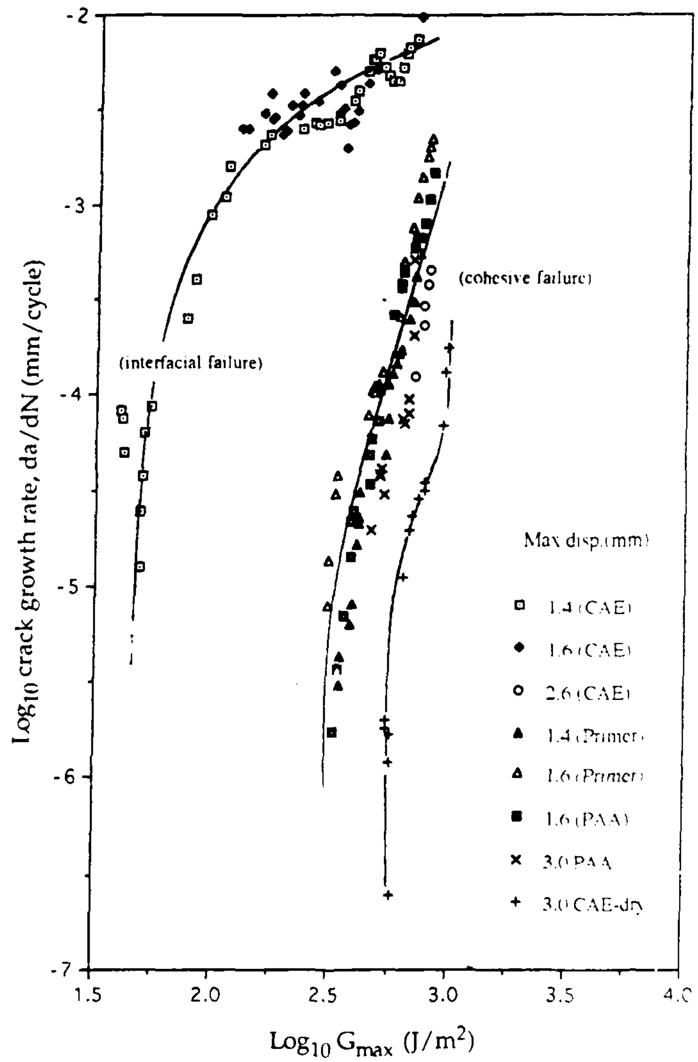

Figure 1 $\quad$ Log_{10} crack growth rate, da/dN, versus $\log_{10} G_{max}$ for aluminium alloy joints bonded with 'AF163' adhesive.

Notes:

CAE:	chromic acid etch pretreament.
PAA:	phosphoric acid anodise pretreament.
Primer:	phosphoric acid anodise pretreament + primer.

Tests conducted in water at 28°C except 'CAE-dry' which were conducted at 55% RH and 25°.